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MECHANICAL PROPERTIES OF THE FLUORIDE-42 POLYMER COMPOSITION WITH ALUMINUM OXIDE SUBMICRONAL PARTICLES

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Abstract— The determination of submicron filler particles (Al_2O_3) concentration influence at the mechanical properties (tensile strength, elongation at break, Martens hardness) of fluoride - 42 composite are considered in that paper. It is shown that the cristallinity of the polymer composition fluoride-42 and oxide alumina submicron particles filler does not change at filler concentrations up to 1%. It is shown that adhesion increasing is contributing to the insignificant increase of the tensile strength and maximum deformation only at low concentrations of filler particles. These mechanical properties are decrease when the filler particles concentration is increase. That's because the inhomogeneities number with local mechanical stress in the interfacial layer are increasing when the filler particles concentration increasing in fluoride-42. (*Abstract*)

Keywords— polymer-matrix composites, fillers, polymer shell, strains, microhardness. (*key words*)

I. INTRODUCTION.

The polymer composite filled by submicron particles has high mechanical properties, such as abrasion resistance, tensile strength, and elongation [1, 2]. The adhesion between submicron particles to the polymer matrix gives the main contribution to the mechanical characteristics of the polymer composition at a low concentration of these particles (no more than 1% by weight). The polymer composition mechanical characteristics with an amorphous and partially crystalline structure are different when the adhesion value between submicron particles and a polymer matrix is the same. It is because the reason of such polymer composition destruction is structural inhomogeneities and interfacial regions, including the interface between filler particles and polymer crystallites [3, 4]. The adhesion value between the filler particles to the polymer matrix is influence on the size of the interfacial region and it mechanical properties [2, 5]. The polymer composite

mechanical characteristics with a partially crystalline structure and an amorphous structure have a different change on the adhesion value change between submicron particles and polymer matrix. So, such polymer composite mechanical properties should depend on the filler particles concentration. The determination of submicron filler particles concentration influence at the mechanical properties (tensile strength, elongation at break, Martens hardness) of fluoride - 42 composite is the purpose of that paper.

II. SAMPLE PREPARATION TECHNIQUE.

The submicron filler particles mass concentration in the polymer matrix was not more than 1% in the experiment investigations. The aluminum oxide (Al_2O_3) submicron particles were used. That submicron particles were production by "Plasmotherm" (Product number: PL1344281) and it was mixture of δ and θ phase with size range 40÷ 190nm. The fluoride grade (F-42) polymer matrix with cristallinity structure was used in experiment investigation. Submicron particles have been activated by polymer shell formation on their surfaces [2]. Polystyrene was used as a polymer for the shell.

The approach to the formation of a continuous polymer shell on the surface of submicron particles in multiphase gas flows was based on the following processes [2].

- Formation of a styrene vapor stream. The consumption of styrene vapor was regulated within the limits of 30–100 mg/s, with an error no more than 5%, by changing the electric power supplied to the heating element of monomer evaporator [2].

- Formation of a two-phase gas flow of submicron Al_2O_3 particles and their subsequent dispersion. The consumption of submicron particles were in the range of 1–

40 mg/s with an accuracy of ± 0.5 mg/s [6]. The speed of the two-phase flow was varied within the range of 1–3 m/s with an accuracy of ± 0.5 m/s. Particle agglomerates were dispersed by their charging in the field of a unipolar corona discharge of atmospheric pressure.

- Condensation of field dispersed styrene droplets from vapor on the surface of submicron particles was carried out in a mixing chamber.

- Deposition of styrene-coated submicron particles in a bath with water, in which styrene was polymerized for at least 4 h. To increase the polymerization efficiency, the suspension was subjected to an ultraviolet radiation. The water temperature in the experiments was $90 \pm 5^\circ\text{C}$. To prevent agglomeration of the encapsulated particles, the suspension, before measuring the shell thickness, was placed for 10 min in a 40-W ultrasonic bath.

The polymer composition fluoride-42 with aluminum oxide particles preparation method was the same of the samples with encapsulated and non capsulated filler particles. The submicron aluminum oxide particles were precipitate in water suspension at the surfaces of the fluoride-42 powder particles at the temperature no more than $80 \pm 2^\circ\text{C}$. The mass ratio of the submicron particles to the fluoride-42 powder particles was controlled with mistake not more than 10%. The filler particles average concentration in polymer matrix was varied at the range 0.06–1.2% with a relative mistake about 10%. So, the polymer composite fluoride-42 filled by submicron aluminum oxide particles was obtained (table 1): sample 1 - polymer composite with encapsulated filler particles; sample 2 - polymer composite with not encapsulated particles. Polymer composite samples for mechanical testing and their structure investigation were obtained by compression formation method of the plate (115x28x2 mm) followed by obtaining a blade (type 1 according to GOST 11262-80): pressing the powder of the samples in the mold under a pressure of 30 kg/cm² for 300 ± 30 s; samples sintering in the mold at a temperature of $220 \pm 2^\circ\text{C}$ for 30 ± 3 minutes; the samples blades were obtained by stamping using a special metal stamp.

Table 1. Sample parameters.

Samples type	Filler particles mass concentration, %	Volume filler particles concentration, [1/ μm^3]
Sample 0	–	–
Sample 1.1 Sample 2.1	0.06	~10
Sample 1.2 Sample 2.2	0.6	~100
Sample 1.3 Sample 2.3	1.2	~200

The samples structure investigations were obtained by Carl Zeiss Merlin scanning electron microscope. The cleaved surfaces of samples that were investigated were obtained after cooling in liquid nitrogen. These samples were placed on the silicon plate surfaces with upside cleaved surface. The gold film was deposited on the surface of the samples (layer 10 ± 2 nm) to eliminate the charging

of the samples during their analysis in a scanning electron microscope.

The Martens hardness investigation was carried out by DUH-211S Shimadzu ultramicrotester according to ISO 14577-1 using a Berkovich indenter (triangular indenter with an apex angle of 115°). The samples several independent measurements were carried out in different regions and there results was averaged. The indentation force of the indenter was 60 mN, the holding time at maximum load was 28 sec.

The tensile strength measurement and elongation in tension of the samples was carried out in accordance with GOST 11262-80 on a universal tabletop testing machine for physical and mechanical testing of various materials Shimadzu AG-X 50 kN. Test parameters: tensile speed - 20 mm/min; temperature - $23 \pm 2^\circ\text{C}$.

III. RESULTS AND DISCUSSION

Micrographs of the samples filled with encapsulated and not encapsulated Al₂O₃ particles are shown in Fig. 1 (aluminum oxide particles in the samples marked by “+”). There are cavities on the samples cleavages surfaces (samples 2.1–2.3), in contrast to Samples 1.1–1.3 (with encapsulated particles).

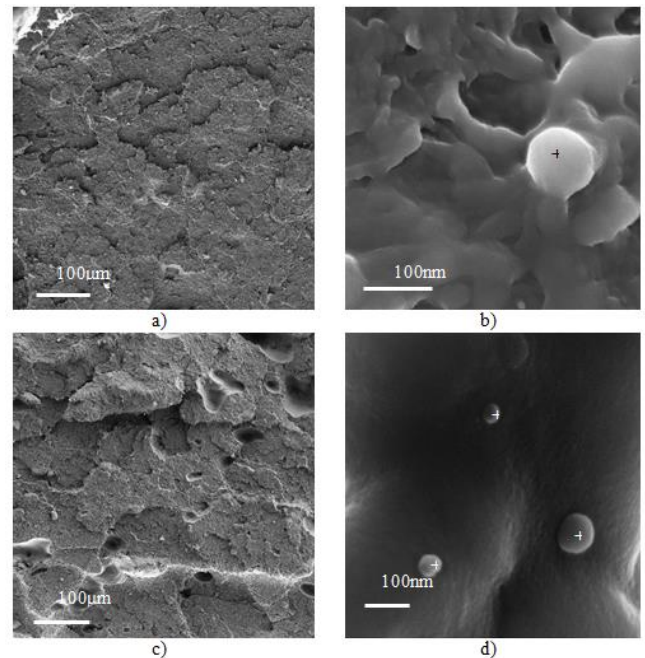


Fig.1. Micrographs of the samples: a,b – Sample 1.3; c,d – Sample 2.3.

The average micro-hardness values (*HMs*) were close for all samples at 37.35 N/mm^2 with a standard deviation of 2 N/mm^2 . The 20 independent measurements of micro-hardness values were carried out in different areas of the each sample for determine the average micro-hardness value (indentation force 60 mN, holding time 28 sec). So, the micro-hardness of the samples does not change when the fillers particle concentration in the fluoride-42 is low, taking into account measurement inaccuracy and variation of sample parameters. The concentration dependences of the tensile strength and the maximum strain, normalised to the corresponding values of Sample 0, are shown in Figure 2.

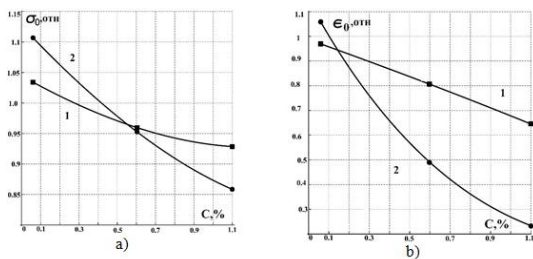


Fig.2. Tensile strength (a) and strain strength (b) as a function of filler particle concentration in the samples: 1 - not encapsulated filler particles; 2 - encapsulated filler particles.

So, the polymer composite (with crystallinity structure) mechanical properties depending of the particle concentration is confirmed by results obtained at Fig. 2. That because of the filler particles number is increasing near the crystallites when the concentration of that particle is growth. Only about 10% of crystallites can interact with filler particles, when the volume concentration of filler particles in polymer matrix is about 10 [$1/\mu\text{m}^3$], characteristic crystallite size is about 170nm with average crystallinity degree $\sim 36\%$. About 100% of crystallites can interact with filler particles when volume concentrations of that particles is above $\sim 200 [1/\mu\text{m}^3]$.

IV. CONCLUSION

So, the nature of the polymer composite with crystallinity structure mechanical properties are different from such properties of polymer composite with amorphous structure, when the dispersed filler particles are used. The tensile strength and maximum deformation of polymer composite (fluoride-42) with crystallitic structure are decrease when the submicron filler particles adhesion to polymer matrix is increasing. There are not changes of fluoride-42 crystallinity when the filler concentration is less than 1%. The number of interfacial areas between the filler and crystallites are increasing and their sizes are change [7] when filler particles concentration is increasing. This is determining the polymer composite mechanical properties decreasing. This can be explained by the mechanical stress gradient increasing at the interfacial areas.

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REFERENCES

- [1] Vasilev A.P., Struchkova T.S., Nikiforov L.A., Okhlopkova A.A., Grakovich P.N., Shim E.L., Cho J.H. Mechanical and tribological properties of polytetrafluoroethylene composites with carbon fiber and layered silicate fillers //Molecules. 2019. Vol. 24. No. 2. Pp. 224.
- [2] Akhmadeev A.A., Bogoslov E.A., Danilaev M.P., Klabukov M.A., Kuklin V.A. Influence of the Thickness of a Polymer Shell Applied to Surfaces of Submicron Filler Particles on the Properties of Polymer Compositions. //Mechanics of Composite Materials. 2020. Vol. 56. No. 2. Pp. 241-248.
- [3] Panin V.E. Physical Mesomechanics Fundamentals //Physical mesomechanics. 1998. Vol.1. Pp.5-22.
- [4] Astuforov S.V., Sholko E.V., Ovcharenko V.E. Psah'e S.G. Influence of the interphase boundaries properties on the mechanical characteristics of cermet composites // Physical mesomechanics. 2014. Vol.17. Pp.53-63.

- [5] Mittal V. (Ed.). Surface modification of nanoparticle and natural fiber fillers. John Wiley & Sons. 2015.
- [6] M.P. Danilaev, N.V. Dorogov, F.A. Karamov, V.A. Kuklin, A.V. Pushkareva Investigation of the characteristics of a device forming a low-speed two-phase gas flow with submicron-size solid particles //Nauch. Tekhn. Vest. Povolzhja. 2019. No.3. Pp.74-77. [In Russ.]
- [7] Saqib S., Rafiq S., Muhammad N., Khan A. L., Mukhtar A., Mellon, N. B., et.al. Influence of interfacial layer parameters on gas transport properties through modeling approach in MWCNTs based mixed matrix composite membranes //Chemical Engineering Science. 2020. Vol. 218. P.115543.